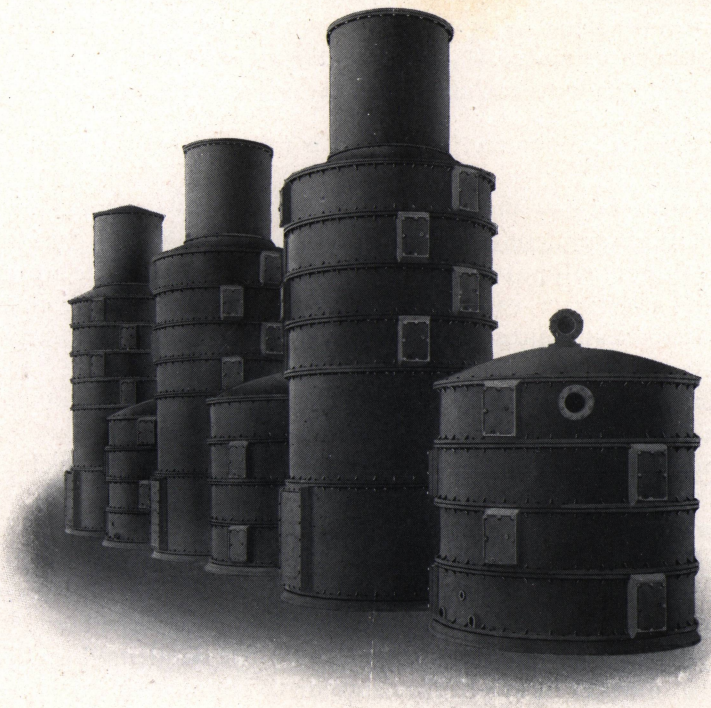
THE WESTERN GAS CONSTRUCTION CO.

GAS ENGINEERS AND BUILDERS

SAN FRANCISCO FORT WAYNE, IND. NEW YORK

Bulletin No. 203.

Ammonia Stills and Concentrators.



Three 84 inch Dia. Fixed and Free Ammonia Stills,
Capacity for 3000 tons Coal Carbonized per diem,
as erected in our Works before Shipment.

AWARDED

GRAND PRIZE AND GOLD MEDAL,

UNIVERSAL EXPOSITION, ST. LOUIS,
1904.

Properties of Ammonia

Ammonia, NH_3, is a colorless gas of pungent odor and sharp alkaline taste. Its specific gravity is .5967 as compared to air and 8.5 when compared to hydrogen.

Its chemical formula NH_3 signifies that it is composed of seventeen parts nitrogen and three parts hydrogen by weight, one part N to three parts H by volume.

By removal of heat or application of pressure, it may be condensed to a mobile colorless liquid boiling under atmospheric pressure at 33.5°C=37°F. Figure 1 shows the curve of its vapor tension from which the pressure necessary to liquify it at any temperature may be read off.

In the ordinary sense, NH_3 is not combustible, but when mixed with hydrogen will burn to water H_2O with liberation of nitrogen.

The solubility in water is shown in the curve in Figure 2.

Commercial Ammonia Products.

As an article of commerce, ammonia is most commonly met with in the following forms:

1. Concentrated gas liquor—A solution of the impure sulphides and carbonates of ammonia, containing more or less free NH_3.

2. Ammonium sulphate, $(NH_4)_2SO_4$, frequently made at gas works. Used largely as a fertilizer, especially in Germany in sugar beet culture. Solutions of ammonium sulphate are used for rendering curtains fireproof. Also as a raw material for the manufacture of other compounds.

3. Ammonium chloride sal-ammoniac NH_4Cl used in pharmacy, in the dye industry and in rust cements, electric batteries, etc.

4. Ammonium Carbonate. Used in scouring wool, in dyeing; as smelling salts, as a baking powder, etc.

5. Aqua Ammonia. Ammonia water—a solution of NH_3 in distilled water. Used in the various arts, as a household cleaning agent, etc. The concentrated aqua ammonia is largely the basis for the manufacture of anhydrous ammonia used so extensively in refrigerating machinery.

6. Anhydrous ammonia, liquid ammonia, the condensed gas NH_3. This comes on the market in steel tubes similar to those used for liquid carbonic acid, and is used for refrigerating purposes.

Ammonia from Coal Gas

Although only about five pounds ammonia is recovered per ton of coal carbonized, the destructive distillation of coal is the principal source of the compound. Its recovery is, therefore, of interest to every gas man.

The crude ammoniacal liquor from the hydraulic main, condensers, washers and scrubbers conveyed to the ammonia well, is a comparatively "weak liquor" averaging from 4 to 16 oz., or 1½°T to 7°T, depending on efficiency of washing and scrubbing apparatus.

Distillation of Ammonia

This necessitates, in nearly all cases, the concentration of the liquor, since freight rates are usually too great to warrant delivery of crude liquor at chemical works. In most cases, nothing beyond this is aimed at, but in

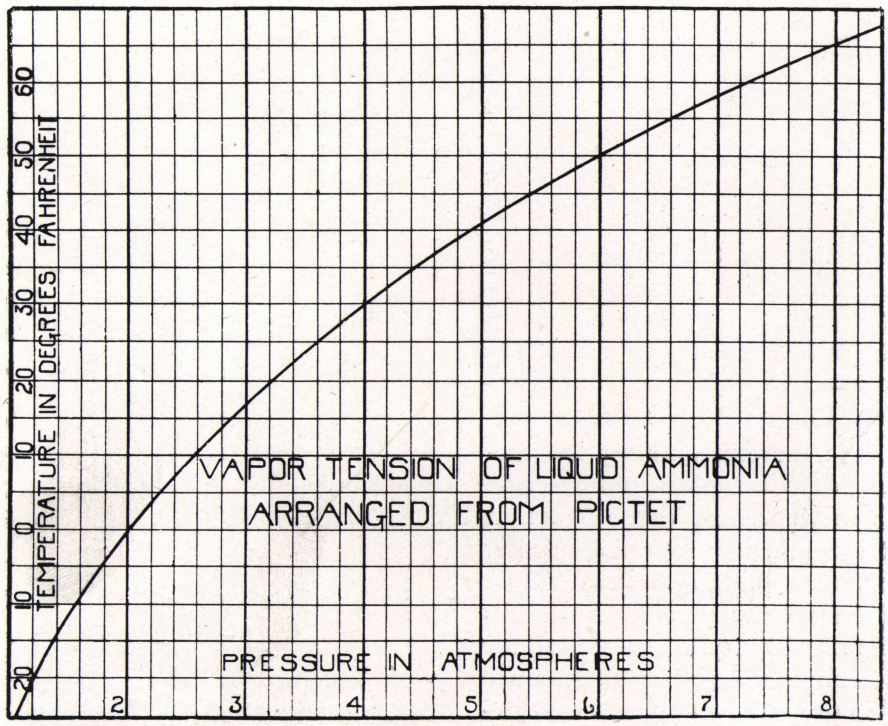

VAPOR TENSION OF LIQUID AMMONIA
ARRANGED FROM PICTET
TEMPERATURE IN DEGREES FAHRENHEIT
PRESSURE IN ATMOSPHERES
20 10 0 10 20 30 40 50 60
2 3 4 5 6 7 8

Fig. 1.

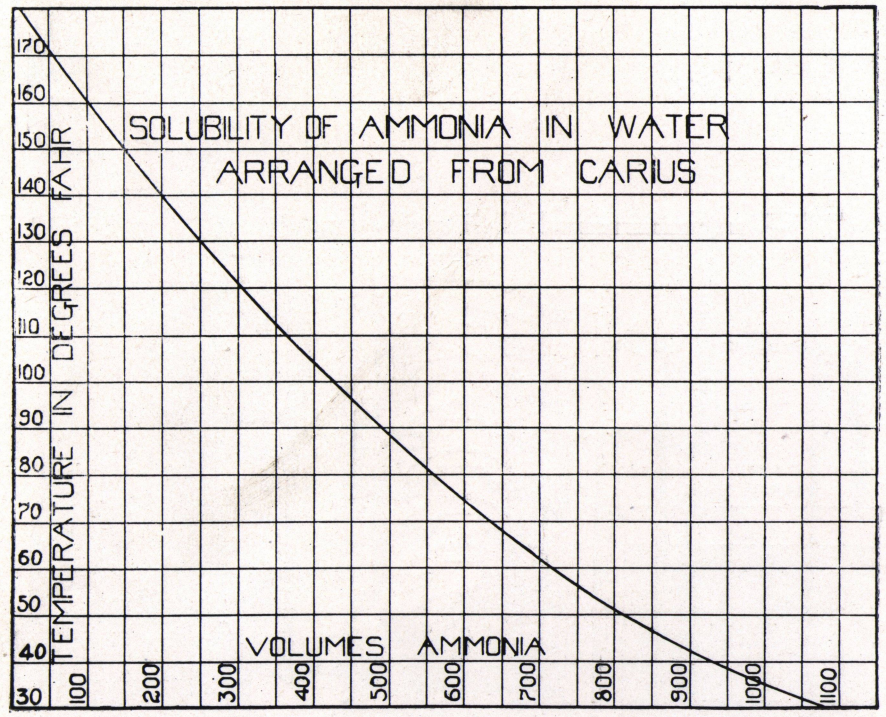

SOLUBILITY OF AMMONIA IN WATER
ARRANGED FROM CARIUS
TEMPERATURE IN DEGREES FAHR
VOLUMES AMMONIA
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30
100 200 300 400 500 600 700 800 900 1000 1100

Fig. 2.

a number of the larger plants; sulphates, aqua or even anhydrous ammonia may be turned out as a finished product at the works. In any case, concentration is necessary as a first step.

It is, therefore, mainly the purpose of this bulletin to describe the arrangement and operation of our machine for this purpose, and only passing notice will be given to the production of sulphate and aqua ammonia, for the manufacture of which we are also prepared to furnish well designed special apparatus as described in our special bulletins.

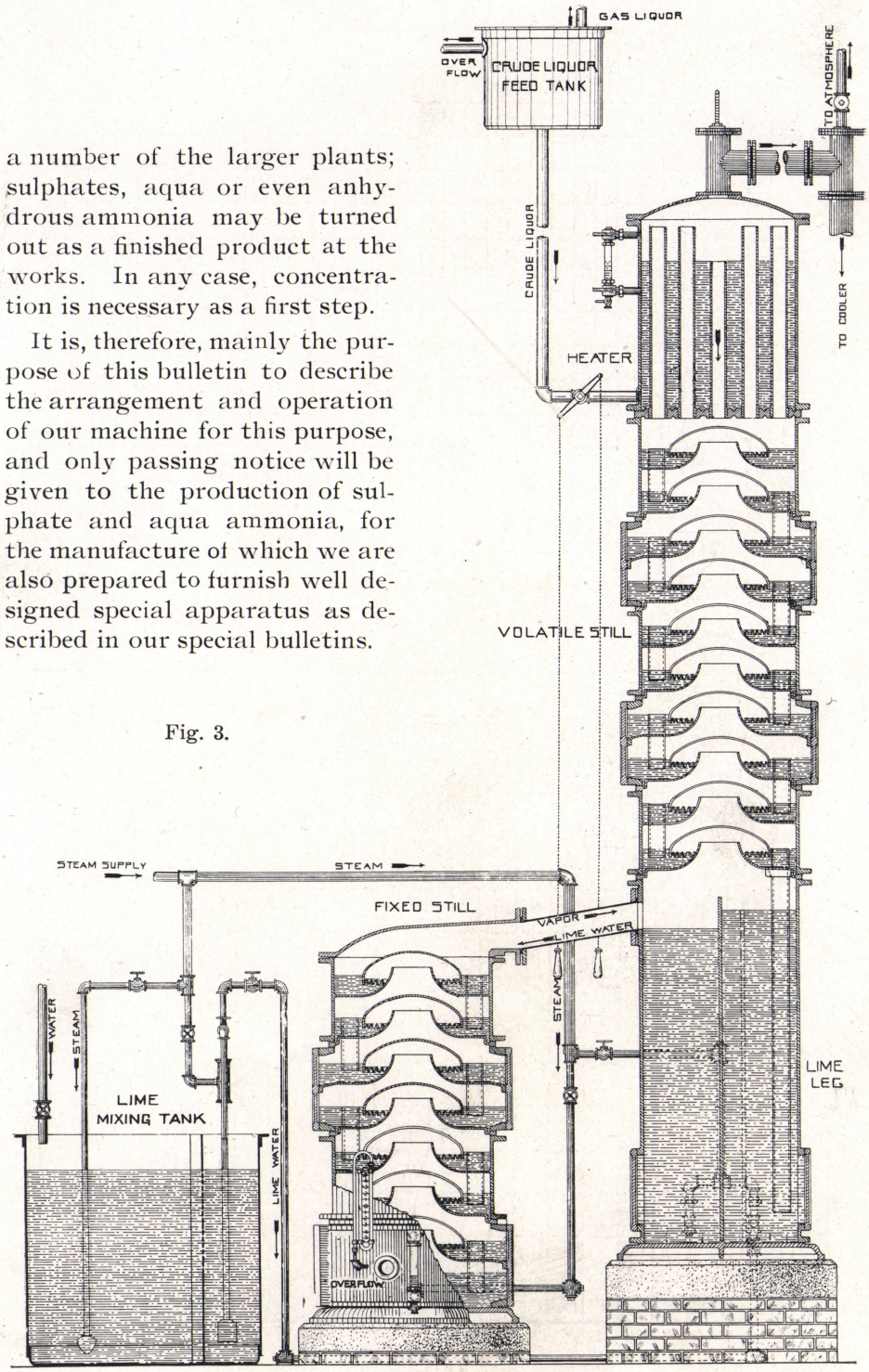


Fig. 3.

Gas liquor is a mixture of various salts of ammonia, some of which are volatile with steam, while others are "fixed" or stable at the boiling point of water. The recovery of these fixed salts necessitates their neutralization by lime. In many small plants it has been considered that this fixed ammonia does not pay for the extra attention and greater first cost of apparatus, and ammonia concentrators have been built to recover only free ammonia. Such an apparatus could be secured by placing our Volatile Still on base below lime leg, changing drain connection to its bottom section and leaving out the Fixed Still, Lime Leg and Lime Mixer. It seems, however, that if it will pay to save 85 percent of the ammonia, it will pay to save all, and even in small works the machine should be so arranged.

In view of recent cases in which gas companies have purchased such *free* ammonia concentrators at what appeared a more favorable price, only subsequently discovering they had simply bought an incomplete apparatus, we desire to state with our recommendations above, that *if it pays to save 85 per cent. of the ammonia it pays to save it all.* We are prepared to sell and erect the simple Volatile Still *at proportionate price of the complete apparatus* where such an apparatus is *specifically requested.*

Description of Valatile and Fixed Ammonia Still.

The apparatus illustrated shows the arrangement of a still of medium size, but the internal arrangement is the same in all, from 24″ to 84″ in diameter. The 18″ machine operates on the same principle, and differs only in that there are somewhat fewer parts.

Heater

For proper understanding of the action of the still, we may trace the flow of liquor from the cistern through the apparatus. An all-iron pump lifts the liquor to an overhead Feed Tank from which it passes through a graduated cock to the heater, where it is preheated by vapors rising through cast-iron pipes fitted by ground joints into the diaphragm which separates this unit from the first section of the still.

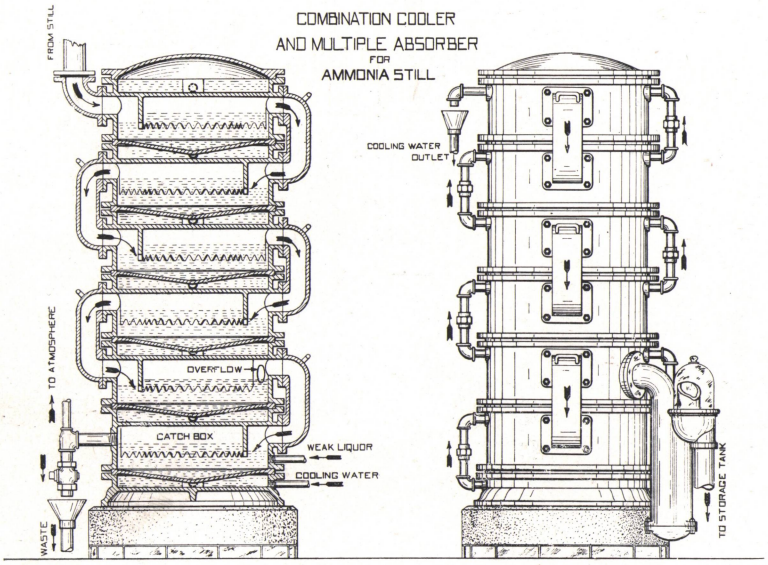


Fig. 4.

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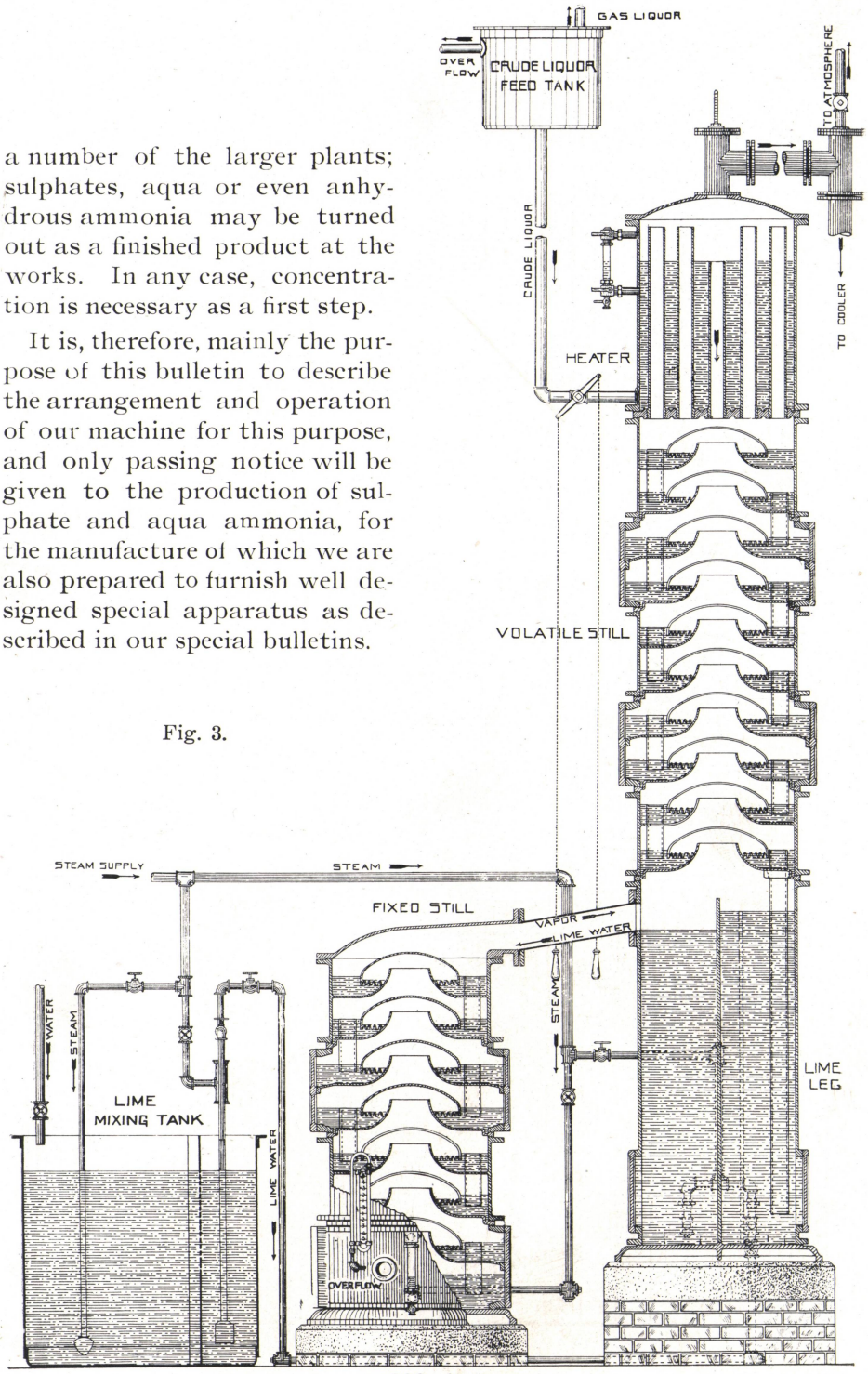

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Heater

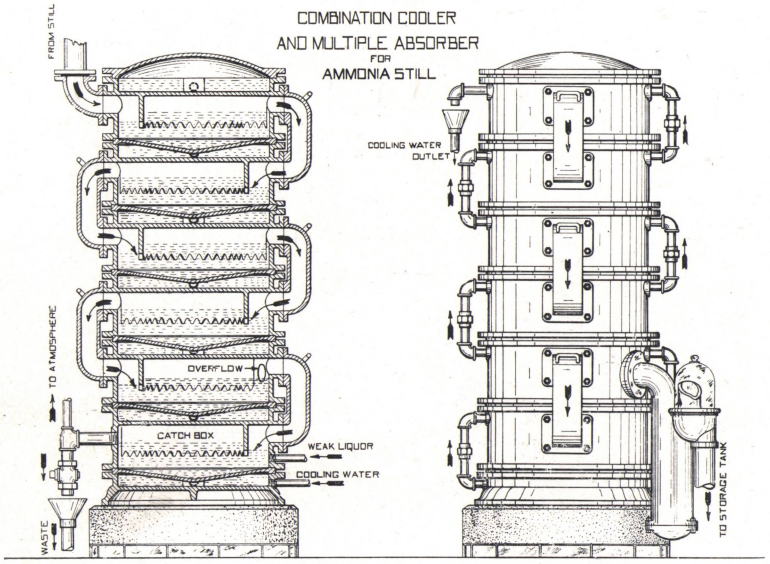


Fig. 4.

The liquor overflows down the central one of these tubes, which is somewhat shorter than the others, into the top chamber of the Volatile Still, which consists, as shown by illustration, of a series of chambers provided with baffles and overflows. The liquor, in descending from chamber to chamber, is brought into intimate contact with the hot rising vapors and reaches the lower section completely freed of its volatile ammonia. The liquor then passes through the long dip pipe down into the Lime Leg, where it is brought in contact with lime just as it enters at bottom of still, The Lime Leg must be of ample capacity, as the *chemical action* necessary is one *requiring time contact*. The arrangement shows how combined liquor and milk of lime are made to traverse the entire length of Lime Leg twice before overflowing to the Fixed Still, thereby insuring excellent, extended and intimate contact between lime and fixed salts. Steam inlets are provided in base of lime leg on each side of the vertical diaphragm, both for the purpose of increasing temperature at this point as well as stirring up the lime mixture, and intimately mixing the liquor with same.

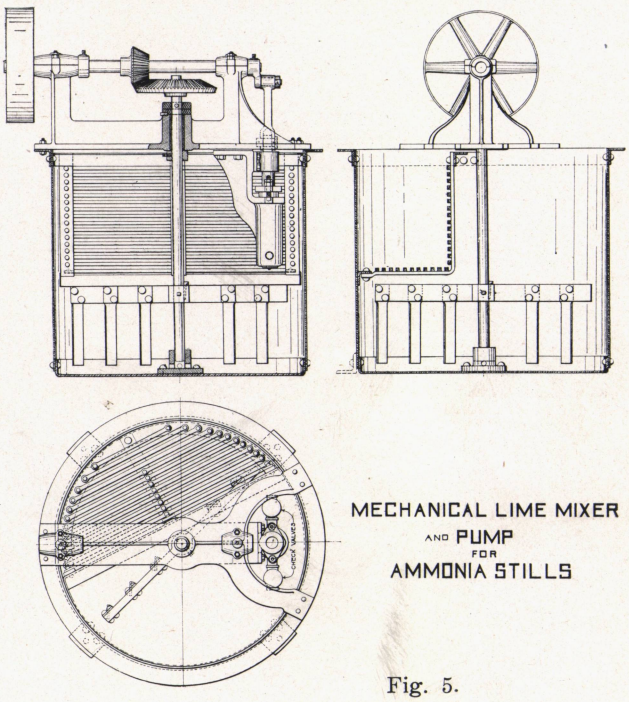
Volatile Still.

Lime Leg or Mixing Chamber.

The liquor and lime water finally overflow into the top section of the Fixed Still and down through a series of chambers identical in construction with those of the Volatile Still. When the bottom section is reached, the waste liquor passes to the sewer and contains only the faintest, if any, trace of ammonia.

Fixed Still.

The design of our stills is the result of several years experience and gradual development by extensive experiments to secure every feature of convenience, simplicity and ease of operation *with maximum efficiency* fully testified to by the extremely low wastes, the control becoming only a matter of keeping the supply of steam and small amount of lime constant and sufficient.



MECHANICAL LIME MIXER
AND PUMP
FOR
AMMONIA STILLS

Fig. 5.

The Lime Mixer shown on Figure 3 is the one used on our small and medium sized stills. It consists of a steel tank divided by a perforated diaphragm into two compartments. The lime is slacked in the largest compartment and the milk of lime thus formed flows through the diaphragm and is delivered by a steam jet pump to the still as required.

Mechanical Mixer.

For large machines, our design of Mechanical Line Mixer is shown in Figure 5.

The slacking compartment consists of basket of iron grating attached to the side of the steel tank in which the bulk lime is placed.

The large compartment is supplied with a rotary paddle and a small pump driven from the same shaft.

With this apparatus the water is continuously supplied at a rate sufficient to furnish the lime water to the still. The water level is kept at such a height that the lime from slacking basket is washed down and well mixed by the rotary paddle.

The power is supplied by belt from a motor or small engine.

Both forms of mixer are fitted up complete with the necessary steam and water connections.

As the steam is introduced in the base of Fixed Still and Lime Leg and ascends through the various chambers, the ammonia passes off, and in rising imparts heat to the liquor in the chambers above until at the top the vapors contain only the amount of moisture necessary for saturation at the temperature maintained.

Combination Cooler and Absorber.

The condensation of these vapors permits the absorption of the ammonia forming the "Concentrated Ammonia" in commercial form, and which is secured by our "Combination Cooler and Absorber," as illustrated on Figure 4. The construction is similar to that of the still, but its function being one of condensation for which purpose water is used, independent water compartments are provided between each liquor or vapor section. The vapors entering the top section from the still condense, in their passage downward from section to section absorbing the free ammonia and overflow continually as concentrated ammonia liquor from the special glass covered overflow of the second bottom section, where the absorption of ammonia is completed. Any vapors with gases remaining pass down to the special bottom section, which acts as a "catch box", being supplied with fresh water or weak liquor, which is allowed to flow back to liquor well or can be pumped to Feed Tank.

Any non-absorbable vapors and gases, escape finally through the vent provided in bottom section.

The cooling water is fed as required into bottom water chamber and rises to the successive water cooling chambers above through outside connections.

Independent Cooler ond Absorber.

A different design of "Independent Cooler and Absorber" is shown in Fig. 6. In this design the Cooler or Condenser is mounted above the absorber as shown. The Cooler is constructed on the principle of air and water cooling in such a manner that the vapors entering at the top from the still travel in a spiral motion downwards to the inlet of absorber. A lead pipe for cooling water passes through the spiral in reverse direction and with the exposed outside and inside walls of Cooler effectively cools and condenses the vapors which absorb the ammonia entering the Absorber through thc central dip pipe. The Absorber in this design consists of a plain vessel only, fitted with a lead coil for cooling water and a continuously operating glass covered overflow. A second Absorber constructed as the first is provided, acting as a "catch box," filled with fresh water or weak ammonia liquor.

This arrangement is now generally used for small sized concentrators only, as the Combination Cooler and Absorber previously described presents material advantages by its reduced working pressure and less floor space required.

The strength of the concentrate depends upon the strength and rate of flow of the crude liquor feed and on the temperature maintained at the head of the still. Liquor of any strength may be produced from 15 per cent., or even above 25 per cent. ammonia content, if necessary. Of course, at very high concentration, troubles may be encountered, due to crystallization of ammonium carbonate.

Usually, it is not desirable to produce liquors above 18 per cent., or about 80 oz.

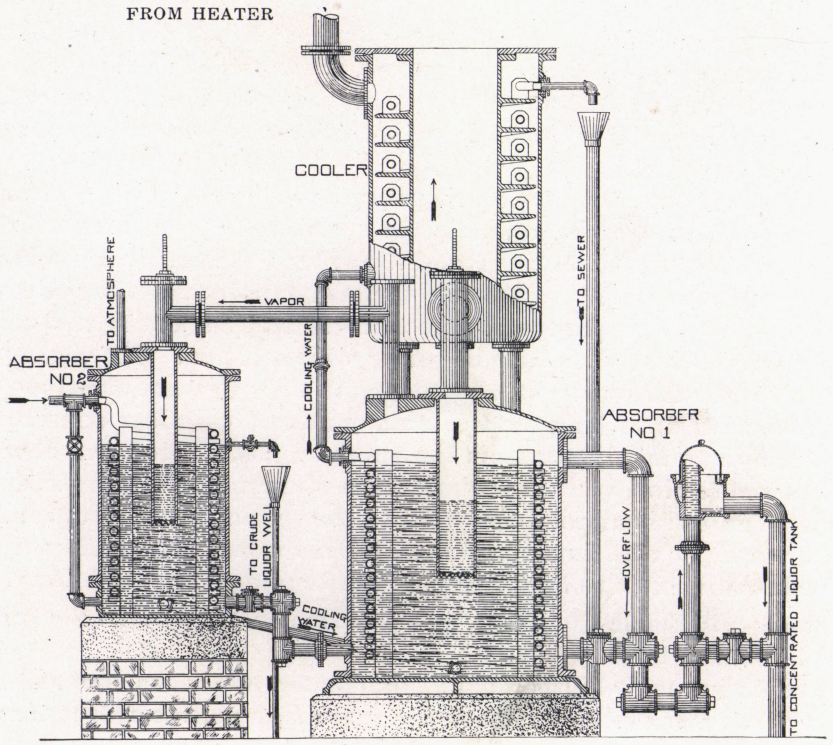
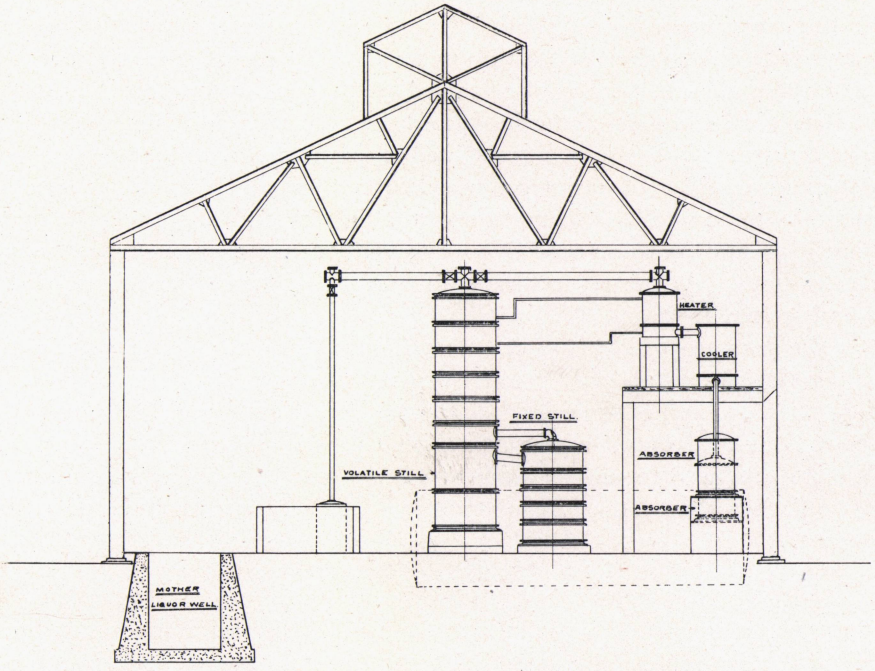


Fig. 6


HEATER
COOLER
FIXED STILL
VOLATILE STILL
ABSORBER
ABSORBER
MOTHER LIQUOR WELL

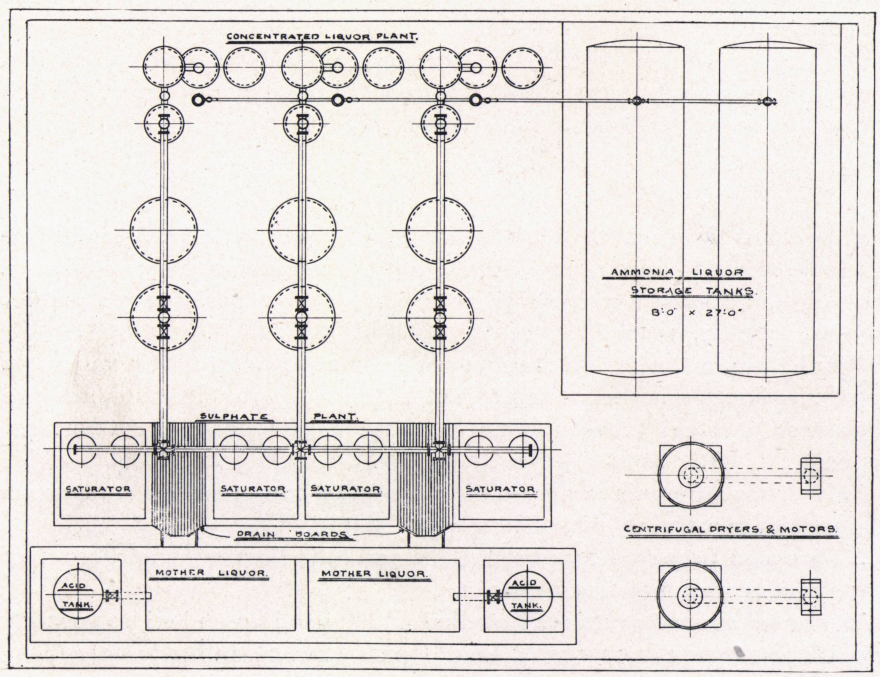

CONCENTRATED LIQUOR PLANT.
AMMONIA LIQUOR STORAGE TANKS.
8'-0" x 27'-0"
SULPHATE PLANT.
SATURATOR
SATURATOR
SATURATOR
SATURATOR
DRAIN BOARDS
CENTRIFUGAL DRYERS & MOTORS.
ACID TANK.
MOTHER LIQUOR.
MOTHER LIQUOR.
ACID TANK.

Fig. 7.

Manufacture of Sulphate of Ammonia.

In the manufacture of sulphate of ammonia it is necessary to substitute a Saturator for an Absorber. The Figure 7 shows a general plan and elevation of a plant for the manufacture of both sulphate and concentrated liquor, the absorbers being on one side of the room and the saturators on the opposite.

Saturator

The Saturator consists of a lead lined compartment, which is supplied with weak sulphuric acid. Vapors from the Still pass into this acid, where the ammonia is absorbed with the formation of the Sulphate $(NH_4)_2SO_4$ the hydrogen sulphide H_2S and carbonic acid CO_2 gases liberated escape through a vent to the atmosphere. As the crystals separate out, they are mechanically removed, dried and sacked.

For the purpose of drying, centrifugal machines are frequently used. In plants where the crystals and mother liquor are drawn from the Saturator to a draining board, a well is provided for the mother liquor, and it is pumped from here back to the saturator, where it is mixed with fresh acid.

We are prepared to submit plans and bids for the construction of sulphate of ammonia plants of any capacity with ordinary or mechanical operating equipment.

Manufacture of Aqua Ammonia.

The manufacture of aqua ammonia usually necessitates a second distillation, although machines have been built for the continuous preparation of pure aqua from weak liquor. In any case, lime sufficient to retain all carbon dioxide and hydrogen sulphide must be added to liquor before distilling.

The vapors are dried by passing through a series of condensers (the condensation passes back to the still), filtered through wood charcoal for the removal of any tarry matters, and finally absorbed into distilled water.

Manufacture of Anhydrous Ammonia.

Anhydrous Ammonia, which is used so largely as a refrigerant, is a pure NH_3 condensed by pressure to a liquid state. Ammonia gas for this purpose may be obtained from a plant similar to that for preparing aqua ammonia, or from pure aqua ammonia.

For the removal of the ammonia gas from aqua ammonia, a vacum pump is necessary. The gas is then compressed to about eight atmospheres pressure, and is stored in steel tubes similar to those used as containers for liquid carbonic acid.

Anhydrous ammonia is purified by repeated compressions and expansions, the impurities remaining behind in a liquid state.

We are also prepared to submit plans and bids for the construction of aqua and anhydrous plants of any capacity.

All our ammonia plants are installed and started up under the special supervision of our chemist in charge of this department, complete instructions being also given for its proper operation. We sell our plants under these conditions.

VALUATION OF GAS LIQUOR.

The valuation of gas liquor depends upon the following reaction between ammonia (NH_3) and sulphuric acid (H_2SO_4). $2\,NH_3 + H_2SO_4 = (NH_4)_2SO_4$, or in other words, 34 parts of NH_3 by weight unite with 98 parts real sulphuric acid to form ammonia sulphate. This reaction takes place whenever these two substances are brought together, regardless of amount of water present. In order to find how much NH_3 is contained in a given amount of gas lqiuor, we have only to know how much of the liquor is taken and the amount and strength of the acid used to neutralize it.

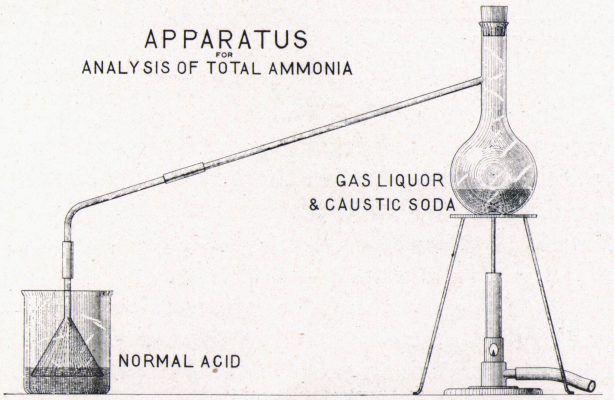
The English ounce strength states these results directly in terms of the H_2SO_4, by weight, required to neutralize one gallon of ammonia liquor, but it is better to calculate these results to per cent. strength since the ratio of ounces to per cent. varies with the specific gravity of the solution.

From the nature of the reaction it is evident that any known strength of acid could be used for this work. It is possible, however, to choose the strength so that much of the calculation is avoided. Thus, it is possible to use such values that ounces may be read off directly on the burette.

It is usual, however, for chemists to employ the so-called normal solution, which contains 49 grammes H_2SO_4 per litre, 1 cubic centimeter of this solution neutralizes .017 grammes NH_3.

For analysis of ammonia liquor, the following apparatus is suggested:

1 distillation flask,
2-50 c. c. burettes and stand,
Glass tubing,
Rubber stoppers,
One 3″ funnel,
1 Beaker (wide pattern),
Solution Normal H_2SO_4,
" " Na_2CO_3,
One 10 c. c. pipette,
One 25 c. c. "
Indicator solution of Methyl orange or cochineal.



Preparing Reagents.

The normal solutions may be made up, if a balance is at hand, by weighing out 53 grammes of pure dry Na_2CO_3 prepared preferably from the bicarbonate by heating to constant weight in platinum crucible and making solution up to exactly 1000 c. c. in graduated flask.

The normal acid may be prepared by titration against this standard carbonate using one of the above indicators. proceeding as described below for analysis of liquor and then diluting the acid to the proper strength. For the average gas works through, it is better to have these solutions made up by a chemical house, such as Eimer and Amend, New York, or Sargent & Company, Chicago.

Measures out 25 c. c. gas liquor into the beaker containing about 50 c. c. distilled water, add a drop or two of the indicator and run in acid drop by drop until the color changes. Suppose 10 c. c. required.

10x4x.017=0.68 Gr. per 100 c.c.

The specific gravity of this solution we will suppose to be 1.01, that is 100 c.c. would weigh 101 grammes

then $\frac{.68 \text{ gr.}}{101 \text{ gr.}} = .673$ per cent.

This method gives only the volatile ammonia since H_2SO_4 does not decompose the more stable or so called fixed salts.

Test for Total Ammonia.

To get the total ammonia, these salts must be decomposed by an alkali and the NH_3 absorbed in the standard acid. The procedure is as follows:

Take 25 c.c. liquor as before and place it together with some water and a stick of caustic soda into the distilling flask of the apparatus set up as in Figure 8. Close the stopper quickly and catch the distillate in say 50 c.c. distilled water containing 25 c.c. acid and a drop or two of indicator. The funnel is to be placed into the beaker to such a depth that the seal will be broken before the acid can be drawn back into the flask. After an hour interrupt the process, neutralize the excess of acid by titrating it with the normal carbonate solution. Suppose 13 c.c. carbonate be used.

$$\frac{4x(25-13)x.017}{101(\text{sp. gr. Liq.})} = 807 \text{ per cent. Total } NH_3$$

.807 per cent—.673 per cent. = .134 per cent. Fixed NH_3.

Calculating Results.

The same method applies to waste liquor and to concentrated liquor, except for the former it is better to use 50 c.c., in the latter 10 c.c. will be quite sufficient. To get these results to terms per 100 c.c. multiply by 2 in the first and by 10 in the second case. The formula for change of degree Twaddell to specific gravity is thus:

1.000 = (2x.005) = 1.01.

To change readings in grammes per 100 c.c. to ounces mutiply by 3.85 or readings in percentage by 3.85x specific gravity. For very dilute liquors the correction for specific gravity does not have much effect but for concentrated ones it is of importance.

From these examples, the relation can be easily seen and it will be possible to use other quantities if more convenient by using the proper factor to reduce reading to parts per 100

In using results based on ounce strength, it must be borne in mind that the British unit differs from our own because of the difference between the U. S. and the Imperial gallon. Confusion is all the more apt to arise, due to this difference, because so many of our text books on gas manufacture are English.

Table showing weight of one Gallon of liquor of from ½° to 26° Twaddell, one gallon water taken at 8.3356 pounds.

Degrees Twaddell.	Sp. Gr.	Pounds Per Gallon.	Degrees Twaddell.	Sp. Gr.	Pounds Per Gallon,
0	1.000	8.3356	13½	1.0675	8.8983
½	1.0025	8.3564	14	1.07	8.9191
1	1.005	8.3772	14½	1.0725	8.9399
1½	1.0075	8.3981	15	1.075	8.9607
2	1.01	8.4190	15½	1.0775	8.9816
2½	1.0125	8.4398	16	1.08	9.0024
3	1.015	8.4606	16½	1.0825	9.0233
3½	1.0175	8.4815	17	1.0850	9.0465
4	1.02	8.5023	17½	1.0875	9.0650
4½	1.0225	8.5232	18	1 09	9.0858
5	1.025	8.5440	18½	1.0925	9.1066
5½	1.0275	8.5648	19	1.095	9.1275
6	1.03	8.5857	19½	1.0975	9.1483
6½	1.0325	8.6065	20	1.100	9.1692
7	1.035	8.6273	20½	1.1025	9.2108
7½	1.0375	8.6481	21	1.105	9.1900
8	1.04	8.6690	21½	1.1075	9.2317
8½	1.0425	8.6899	22	1.11	9.2525
9	1.0450	8.7107	22½	1.1125	9.2734
9½	1.0475	8.7315	23	1.1150	9.2942
10	1.05	8.7524	23½	1.1175	9.3150
10½	1.0525	8.7732	24	1.12	9.3359
11	1.055	8.7941	24½	1.1225	9-3567
11½	1.0575	8.8149	25	1.125	9.3776
12	1.06	8.8357	25½	1.1275	9.3984
12½	1 0625	8.8566	26	1.13	9.4192
13	1.065	8.8774			

We build only first-class plants, installing same complete and in operation under supervision of our men experienced in this department.

Our plants. the result of extended experience, will require a minimum of labor and expense of operation and be a continued source of maximum revenue.

We do not care to build a cheap plant, or plants in which first cost is the only consideration, too many such have been found to be an expensive luxury to operate—too often really expensive as a gift.

Any coal gas company carbonizing over six tons of coal per diem and with proper scrubbing plant can very profitably install a *good* concentrating plant.

TABLE OF SIZES AND CAPACITIES OF AMMONIA CONCENTRATING STILLS.

Size.	Height	Approximate Floor Space.	For Plants Carbonizing.
18″	14′0″	6′x12′	20— 30 tons.
24″	18′0″	7′x14′	60— 75 "
36″	23′0″	8′x16′	100— 150 "
48″	23′0″	9′x18′	250— 300 "
60″	24′0″	10′x20′	400— 450 "
72″	25′0″	11′x23′	600— 650 "
84″	27′0″	12′x25′	800—1000 "

Our ratings are very conservative, being based on the production of four ounces (about 10 per cent.) crude liquor. Plants requiring stills of 36″ or above should with their better scrubbing equipment easily produce liquors of 8-12 oz. This means proportional increase in capacity of the still.